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**United States
Department of
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**Marketing and
Regulatory
Programs**

**Grain Inspection,
Packers and
Stockyards
Administration**

**Federal Grain
Inspection
Service**

Washington, DC

November 1999

Near-Infrared Transmittance Handbook (NIRT)

Program Handbook

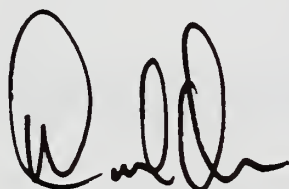
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Near-Infrared Transmittance Testing

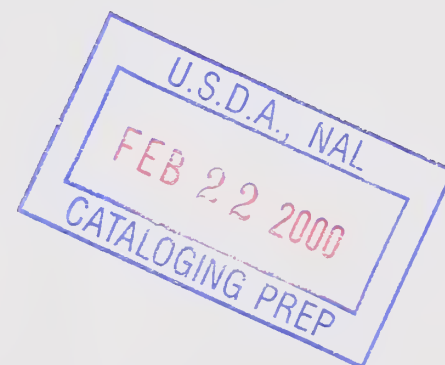
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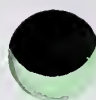
The Near-Infrared Transmittance Testing (NIRT) Handbook has been updated to consolidate instructions for performing tests for wheat protein, soybean protein and oil, and corn protein, oil, and starch. This handbook illustrates step-by-step procedures for making official protein, oil, and starch determinations using the Tecator 1225, 1226, 1227, and 1229 NIRT (whole grain) analyzers. All official inspection personnel authorized or licensed to perform NIRT testing shall reference this handbook for procedures.

This handbook supersedes the NIRT Handbook, dated 8-2-96.



David Orr, Director
Field Management Division





United States Department of Agriculture
Grain Inspection, Packers and Stockyards Administration
Federal Grain Inspection Service

FGIS Issuance Change

CHANGE TO

☐ DIRECTIVE

☐ MANUAL

X ☒ HANDBOOK

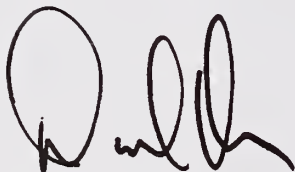
CHANGE NO: 2	TO (No.)	TITLE: Near-Infrared Transmittance Testing (NIRT) Handbook	DATE: 11-22-99
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PURPOSE OF CHANGE:

The NIRT Handbook has been revised to incorporate changes made to the official NIRT testing program since 1996. The revisions include: (1) procedures for analyzing corn for oil, protein, and starch content; (2) the addition of Infratec model 1229 as an approved instrument; and (3) procedures for standardizing field instruments using the pathlength method.

FILING INSTRUCTIONS

Remove NIRT Handbook, dated 8-2-96, and replace with NIRT Handbook dated 10-18-99. Retain Issuance Change Sheet as an aid in verifying handbook content.



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GRAIN INSPECTION, PACKERS AND STOCKYARDS
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WASHINGTON, D.C. 20090-3630

NIRT HANDBOOK
11-22-99

Near-Infrared Transmittance Testing

TABLE OF CONTENTS

CHAPTER	TITLE
1	INTRODUCTION
2	NIRT EQUIPMENT
3	DETERMINING INSTRUMENT ACCURACY
4	SAMPLE PREPARATION AND ANALYSIS
5	NIRT MONITORING PROGRAM



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NIRT HANDBOOK
CHAPTER 1
11-22-99

Near-Infrared Transmittance Testing

CHAPTER 1

INTRODUCTION

Section Number	Section	Page Number
1.1	PURPOSE	1-1
1.2	SCOPE	1-1
1.3	DEFINITIONS	1-1
1.4	RESPONSIBILITIES	1-4
1.5	DISCLAIMER CLAUSE	1-7

Near-Infrared Transmittance Testing

INTRODUCTION

1.1 PURPOSE

This handbook establishes procedures for determining and certifying official protein content in wheat, official protein and oil content in soybeans, official protein, oil, and starch content in corn, monitoring the accuracy of official results, and maintaining near-infrared transmittance (NIRT) equipment accuracy.

1.2 SCOPE

Testing wheat for protein content, soybeans for protein and oil content, and corn for protein, oil, and starch content as "official criteria" is authorized under Section 7(b) of the United States Grain Standards Act (USGSA), as amended. Sections 800.125 and 800.135 of the USGSA permits a review inspection on either official grade/factors or official criteria. When requested, a review inspection for official grade or official factors and official criteria may be handled separately even though both sets of results are reported on the same certificate. All official protein, oil, and starch analysis under the USGSA shall be performed in accordance with procedures prescribed in this handbook and the instrument manual and must be performed only by authorized or licensed employees of the Federal Grain Inspection Service (FGIS) or delegated/designated agencies.

1.3 DEFINITIONS

- a. **Baseline** - Known protein, oil, and starch values determined by the Inspection Systems Engineering Branch (ISE), Technical Services Division (TSD), for the Standard Reference Sample (SRS) sets.
- b. **Bias** - The average difference between NIRT results and the baseline values assigned by ISE to the SRS. To "bias" an NIRT instrument, adjust the instrument's intercept constant by the negative of the amount of the calculated bias.
- c. **Calibration Constants** - The instrument requires a calibration disk containing the necessary calibration constants. The NIRT instrument uses these numerical values to calculate the protein content for the class of wheat being tested, the protein and oil content of soybeans, and the protein, oil, and starch content of corn.

- d. **Calibration Disk** - A Tecator Infratec compatible floppy disk where the NIRT calibration constants, instrument constants, and the instrument operating system are magnetically stored.
- e. **Check Samples** - Samples distributed to official inspection points by ISE for the purpose of comparing field results to ISE results.
- f. **Coarse Foreign Material** - Foreign material found in soybean samples that consists of the following:
 - (1) Whole kernels of corn. Whole kernels of corn are kernels with one-fourth or less of the kernel removed.
 - (2) Cockleburs.
 - (3) Sticks meeting the following criteria:
 - (a) Approximately 1 inch or more in length.
 - (b) Approximately ½ inch or more with a thickness of 5/32 of an inch (width of the largest soybean slotted sieve).
 - (4) Pods (one-half pod or more). If pods contain soybeans, remove the soybeans and return to sample.
 - (5) Other coarse foreign material may include, but is not limited to, corn cobs, large feed pellets, pieces of dirt larger than soybeans, sweet corn, and edible beans that are generally larger than soybeans.
- g. **Collaborative Study** - A study designed to compare constituent values determined by different laboratories.
- h. **Combustion Method** - A chemical analysis used to determine the percent nitrogen in a sample using the AOAC International Method 992.23.
- i. **Constituent** - Compounds for which an analysis is made in a product (i.e., protein in wheat, protein and oil in soybeans, protein, oil, and starch in corn).
- j. **Correlation** - The interdependency of one variable on another (i.e., solvent oil extraction and NIRT oil).

- k. **Instrument Constants** - Several constants (some of which are different for each instrument) entered on the instrument's calibration disk prior to using the disk for official determinations.

The instrument constants that must be entered for each unit include "O" and "P" constants (which characterize the instrument's optical components) and the slope and intercept constants that adjust each instrument for optimum agreement with the standard reference method. No other constants shall be altered by the operator.

Disks containing calibration constants are no longer interchangeable among NIRT units after instrument constants are entered. Each NIRT unit must be used with its own calibration disk.

- l. **Monitor Samples** - Samples selected by a specified process from the officially inspected samples analyzed during a period of time. The official results on these samples are compared to the results obtained by a monitoring office to detect developing protein inaccuracies in wheat, protein and oil inaccuracies in soybeans, and protein, oil, and starch inaccuracies in corn.
- m. **Near-Infrared Transmittance (NIRT) Determination** - A spectrophotometric determination of a sample's constituents by measuring the amount of light transmitted through a sample at specific wavelengths in the near-infrared region of the spectrum.
- n. **Oil** - Lipids (oils and fats) that are liquid at room temperature.
- o. **Pathlength** - With the sample cell installed in the instrument, it is the distance between the two glass windows on opposite sides of the grain stream. One of these windows is on the detector module and the other is on the removable sample cell.
- p. **Polarimetry** - A technique that measures the rotation of the plane of polarized light or the degree of polarized light passing through an optical sample to determine the starch content using the Corn Refiners Association Method A-20.
- q. **Protein** - A naturally occurring complex combination of amino acids joined by peptide bonds that contain the elements carbon; hydrogen; nitrogen; oxygen; sulphur; and, to a lesser degree, other elements.

- r. **Sample Transport Module** - An accessory to the Infratec 1225/1226/1227 Grain Analyzer used in place of the hopper, sample cell, and brush wheel, to analyze small sample portions.
- s. **Solvent Extraction** - A process that uses a non-polar solvent such as petroleum ether to extract the oil from a sample using the FGIS solvent oil extraction method.
- t. **Specified Service Point** - A city, town, or other location where official services are performed by an official agency or FGIS personnel.
- u. **Standard Reference Method** - A standardized chemical method used to determine a constituent value for a sample. FGIS standard reference methods are used to develop the calibration constants used with NIRT instruments for each class of wheat, and for soybeans and corn.
- v. **Standard Reference Samples (SRS)** - A set of samples with established protein values for each class of wheat, protein and oil values for soybeans, and protein, oil, and starch values for corn which are used to maintain NIRT instrument accuracy.
- w. **Standard Slope Settings** - Average slope value used by all instruments within the official system. It is based on the individual slope values for instruments maintained by the TSD and covers grain from multiple crop years.
- x. **Starch** - Any of a group of polysaccharides composed of long-chain polymer of glucose in the form of amylose and amylopectin. It is the chief storage form of energy reserve (carbohydrates) in plants.

1.4 RESPONSIBILITIES

The general responsibilities for the wheat protein, soybean protein and oil testing, and corn protein, oil, and starch programs are as follows:

- a. Responsibilities of the Inspection Systems Engineering Branch (ISE).
 - (1) Maintain the FGIS National Standard NIRT instruments.
 - (2) Develop, maintain, and implement the calibrations for official NIRT instruments. This includes developing the standard slope settings associated with an updated calibration and issuing the standard slope settings used by official NIRT instruments.

- (3) Establish the official SRS protein contents for each class of wheat, protein and oil contents for soybeans, and protein, oil, and starch contents for corn.
 - (4) Develop and distribute SRS sets to FGIS field offices and official agencies.
 - (5) Prepare and distribute check samples (biannual for soybeans, annual for corn) to monitor the accuracy of official testing locations providing soybean protein and oil, or corn protein, oil, and starch testing service.
 - (6) When necessary, review NIRT procedures at FGIS field offices and official agencies.
 - (7) Monitor the accuracy of official wheat protein, soybean protein and oil, and corn protein, oil, and starch testing locations.
 - (8) Initiate, conduct, and report collaborative and special studies as needed.
 - (9) Provide technical support and training to official inspection personnel in matters relating to NIRT determinations, maintenance, service, and repair.
- b. Responsibilities of the Board of Appeals and Review (BAR).
- (1) Provide maintenance, service, and repair for federally owned instruments.
 - (2) Coordinate requests for Board appeal inspection services.
 - (3) As applicable, issue certificates and assess fees for Board appeal inspection services.
- c. Analytical, Reference, and Testing Services Branch (ARTS). Maintain the FGIS standard reference laboratories. Establish the official wheat protein content, soybean protein and oil contents, and corn protein, oil, and starch contents for calibration samples and slope samples.
- d. Responsibilities of FGIS Field Office Managers.
- (1) Select an NIRT coordinator to serve as the primary contact within the respective circuit and to ISE.

- (2) Perform original, reinspection, and appeal wheat protein, soybean protein and oil, and corn protein, oil, and starch inspection services as applicable.
- (3) Retrieve and distribute the wheat protein control charts and collaborate with ISE in monitoring specified service points within the circuit.
- (4) Routinely review NIRT procedures in the respective area of responsibility.
- (5) When necessary, inform ISE of problems detected in respective areas of responsibility and initiate appropriate corrective action.
- (6) Provide technical support and training to official inspection personnel.
- (7) Assist ISE in conducting collaborative and/or special studies.
- (8) Assure that official agencies select and forward wheat protein monitoring samples to the appropriate field office and/or ISE.
- (9) Mail file samples for Board appeal inspection services to the BAR using a red mailing tag. Write the words "Protein Board Appeal" in the "Remarks" section of the grain sample ticket and on the back of the mailing tag.
- (10) On a case-by-case basis and with the approval of the Director, Field Management Division, provide original wheat protein, soybean protein and oil, and corn protein, oil, and starch testing service in areas where agencies are unable to arrange for adequate services (see Section 1.4e(2)).
- (11) Send soybean and corn samples, upon request, to ISE for use in producing check samples and evaluating calibration performance.
- (12) Test soybean and corn check samples and submit results to ISE within 10 days of receipt.

e. Responsibilities of Agency Managers.

- (1) Coordinate and maintain a wheat protein, and/or soybean protein and oil, and/or corn protein, oil, and starch testing program in assigned geographic areas.

- (2) Perform original and reinspection NIRT wheat protein, and/or soybean protein and oil, and/or corn protein, oil, and starch inspection services and forward file samples for appeal to the FGIS field office or ISE. In some instances, the demand for the wheat protein, soybean protein and oil, or corn protein, oil, and starch testing service may not warrant the purchase of NIRT equipment by an agency. If this occurs, the agency must survey industry representatives, determine the need for service, notify FGIS of their findings, and locate a mutually agreeable agency to provide the service. If an agency cannot arrange for adequate service, FGIS will determine on a case-by-case basis whether FGIS will provide original wheat protein, and/or soybean protein and oil, and/or corn protein, oil, and starch testing service.
- (3) Select and forward wheat protein monitoring samples to the appropriate FGIS field office and/or ISE.
- (4) Test soybean and corn check samples and submit results to ISE within 10 days of receipt.
- (5) Routinely review the NIRT operator's procedures at testing sites within the assigned geographic area.
- (6) Provide technical support and training to NIRT technicians.
- (7) Assist ISE/FGIS field offices in conducting collaborative and special studies.
- (8) Notify the monitoring FGIS field office concerning NIRT related problems and initiate follow-up action.
- (9) Maintain complete work records.

1.5 DISCLAIMER CLAUSE

The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned.



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NIRT HANDBOOK
CHAPTER 2
11-22-99

CHAPTER 2

NIRT EQUIPMENT

Section Number	Section	Page Number
2.1	OFFICIAL EQUIPMENT	2-1
2.2	CALIBRATION	2-1
2.3	NIRT TESTING FACILITIES	2-2
2.4	SETUP	2-4
2.5	EQUIPMENT MAINTENANCE	2-5

CHAPTER 2

NIRT EQUIPMENT

2.1 OFFICIAL EQUIPMENT

Tecator - Infratec Models 1225, 1226, 1227, and 1229 are approved for official wheat protein, soybean protein and oil, and corn protein, oil, and starch determinations. All approved models currently being manufactured will be equipped with a hard drive. Hard drive units will need to have the official calibrations loaded into the instrument prior to testing.

2.2 CALIBRATION

- a. FGIS approved calibrations are required for official NIRT instruments.
- b. The ISE is responsible for maintaining and updating calibrations for official NIRT instruments. Contact ISE to obtain the approved calibration disk, standard slope settings, and reference sample sets.
- c. ISE provides one disk for each NIRT instrument. This disk will contain all FGIS approved calibrations (wheat, soybeans, and corn) and the current Tecator operating system software.
- d. New instruments must have their monochromator checked and the wheat sample cell pathlength standardized between 18.65 - 18.85 millimeters. Standardized instruments are issued the standard slope settings for each grain type they test. Analyze the appropriate Standard Reference Samples (SRS) twice and bias the instruments using the Level II tolerances before the instrument is used for official testing. Major instrument repair (e.g., monochromator replacement) will require that the monochromator be checked before official testing can resume. Replacing the sample cell will require that the pathlength be measured and the SRS tested to check the bias before official testing can resume. Operators must use the standard slope settings, SRS sets, and baseline values provided by ISE.

Note: Infratec 1229s that are equipped with the variable (adjustable) sample cell do not need to standardize the pathlength. They will use the software assigned pathlength of 18.00 millimeters for wheat and/or 30.00 millimeters for corn and soybeans.

- e. ISE will maintain a master list of all NIRT instruments in the official system and their approved calibration information. Upon request, ISE will forward a list of all instruments and their approved calibration information to the appropriate FGIS field office or official agency.
- f. FGIS field office and official agency managers shall verify the following:
 - (1) The serial number on the calibration disk corresponds to the serial number of the instrument;
 - (2) Individual instrument constants ("O", "P", slope, and intercept) have been correctly entered onto the calibration disk and none of the other instrument constants have been altered;
 - (3) The calibration name is identical to that currently specified by ISE;
 - (4) The calibration disk is the current version approved by ISE;
 - (5) Slope values agree with ISE records; and
 - (6) NIRT instruments are configured by FGIS calibration to give wheat protein readings corrected to a 12 percent moisture basis (mb), soybean protein and oil readings corrected to a 13 percent mb, and corn protein, oil, and starch readings corrected to a dry matter basis directly without further calculations.

2.3 NIRT TESTING FACILITIES

Equipment location and environmental factors can affect the performance of NIRT equipment.

- a. Location of Equipment. NIRT instruments must be placed in a location conducive to a dust-free and stable environment. If the NIRT instrument is not located in its own room, all other dust-emitting devices located in the same room must be operated with a functional dust collection system. The NIRT instruments must be protected from drafts, heating and cooling vents, and windows. Also, a vibration-free table is recommended to support the NIRT instrument.
- b. Environmental Requirements. The space and facilities required to perform official NIRT determinations must meet the specifications outlined below:

- (1) Temperature. Temperature affects the stability of NIRT instruments. Each testing site shall install a thermometer near the NIRT instrument(s). **The temperature of the room where official testing occurs must be maintained between 60° and 80°F (16° and 27°C).** Official testing shall be suspended if the room temperature is outside the acceptable range. Once the temperature is restored to the acceptable range, check instrument accuracy using the SRS set and, if necessary, bias adjust the instrument.

If the room temperature changes by $\pm 5^{\circ}\text{F}$ (2.5°C) or more from the temperature recorded during the daily instrument check, retest the SRS and, if necessary, bias the instrument.

- (2) Relative Humidity. **Relative Humidity (RH) must be kept between 20 and 75 percent.** Each testing site shall install a hygrometer (calibrated to ± 3 percent RH) near the NIRT instrument(s). When the laboratory's RH is outside of the acceptable range, retest the SRS and, if necessary, bias adjust the instrument based on LEVEL-I tolerances. Once the laboratory's RH returns to the acceptable range, the SRS need to be retested **only if** a bias adjustment was made while the RH was outside the acceptable range. If necessary, bias adjust the instrument based on the LEVEL-I tolerances. SRS sets collected when the RH is outside of the acceptable range may not be used for the LEVEL-II and higher tolerances. All LEVEL tolerances are listed in section 3.2 for wheat, section 3.3 for soybeans, and section 3.4 for corn.

FGIS field offices shall periodically check individual testing location(s) hygrometers using a battery powered psychrometer. Before checking the hygrometers, check the psychrometer thermometers when both are dry to determine if they are in agreement. Then check hygrometers against the psychrometer and apply a tolerance of ± 5 percentage points. Repair or replace hygrometers which deviate from the psychrometer by more than 5 percentage points.

- (3) Power Supply. The power for all NIRT instruments shall be supplied by a 120 ± 10 VAC/15-20 amp dedicated circuit. A maximum of two electronic instruments (i.e., NIRT, NMR or Hardness Tester) plus their associated printers and/or computers may be placed on one dedicated circuit. No other equipment shall be used on the circuit.

NOTE: If a dedicated circuit cannot be provided, a computer grade uninterruptable power supply (UPS) with line conditioner is an acceptable alternative. Before purchasing or installing an UPS, written verification must be obtained from the NIRT instrument manufacturer that the specific model of UPS proposed is compatible with the NIRT instrument. A copy of the verification letter must be kept on file.

A power line conditioner is recommended for use if line voltage variation is a suspected problem. Before purchasing and installing a voltage regulation device, contact the instrument manufacturer to determine which device is best suited for this purpose.

An NIRT instrument may be turned off if it will not be used for at least 8 hours. After turning the instrument on, it must be allowed to warm up at least 15 minutes before testing. Outliers in the "A" or "B" position of the outlier code may be indicated as a result of insufficient warm up.

- (4) Smoke and Dust. Post "**NO SMOKING**" signs in the testing area. Follow good housekeeping practices to maintain a clean and dust-free environment. Use a vacuum cleaner or brush for proper laboratory cleanup. Do not use compressed air for cleanup purposes.

2.4 SETUP

Official testing agencies and FGIS field offices must observe certain guidelines when establishing new laboratories, placing new equipment on-line, or relocating NIRT equipment.

- a. Laboratory Setup. Upon request, ISE will assist official agencies in planning and preparing laboratories for official NIRT testing. Official agency managers must notify the appropriate FGIS field office manager concerning plans for a new laboratory and provide a diagram of the proposed design. The diagram should contain the proposed locations of NIRT equipment, location of major inspection equipment, and description of the power supply. Any additional information regarding the laboratory setup or equipment should also be included. The monitoring field office manager will forward a copy of all submitted information to ISE for review. Upon receipt, ISE will review the information and make recommendations to the official agency manager and monitoring FGIS field office manager to facilitate the laboratory setup.

- b. Equipment Setup. Official personnel shall notify ISE and the appropriate field office when new NIRT instruments are purchased. ISE will provide the necessary samples and instructions to check the accuracy of the instrument(s). Contact ISE as soon as possible because the checkout process may take several days to complete.

When an NIRT instrument is moved to a new location, the instrument must be allowed to reach temperature equilibrium with its environment before performing official tests. Generally, the instrument should sit for at least 2 hours before use after being moved. If the instrument might have been subjected to extreme temperatures during shipment, allow the unit to sit overnight in the new location before operating it.

- c. Sample Transport Mechanism. If a testing location must use the "sample transport mechanism" to handle smaller sample sizes, contact ISE for requirements and setup instructions.

2.5 EQUIPMENT MAINTENANCE

- a. General.

- (1) Using a brush or cloth, dust out the sample hopper and path each day.
- (2) Replacement lamps for the instrument are expensive and, therefore, the lamp life should be extended as long as possible. Turning the lamp on and off frequently decreases its life. Turn the instrument off only if it will not be used for a period of 8 hours or more.
- (3) Major instrument repair (e.g., monochromator replacement) will require running the monochromator check before official testing can resume. To minimize "OUT OF SERVICE" time, notify ISE to schedule the monochromator check after the instrument has been repaired.

- b. Repair of FGIS-owned NIRT Equipment.

- (1) Repair and service of FGIS-owned instruments are coordinated by FGIS personnel presently located at the FGIS Technical Center in Kansas City, Missouri.

(2) BAR and ISE personnel are assigned to assist field office personnel in: (a) maintaining instruments, (b) performing diagnostic tests needed to verify acceptable performance, and (c) performing circuit board replacement when required.

(3) Repair Procedures.

(a) If an NIRT instrument malfunctions, the designated field office NIRT coordinator should contact the BAR at (816) 891-0435 to report the problem.

(b) The NIRT coordinator should be prepared to answer all questions regarding the symptoms of the failure (error codes, erroneous readings, malfunctioning display, etc.) and to perform diagnostic tests while maintaining telephone communications with the BAR.

(c) The BAR will take one of the following actions:

1 If the NIRT instrument is determined to be field repairable, the BAR will coordinate the shipment of replacement parts (boards, etc.) to the field office.

2 If it is not field-repairable, the BAR will authorize return of the instrument to Kansas City. The field office will be responsible for shipping costs from the field to the BAR. If necessary, a replacement instrument will be furnished to the field office by the BAR. A written summary of the malfunction should be included with the field unit. Send the unit to:

USDA, GIPSA, FGIS, TSD
10383 N. Executive Hills Blvd.
Kansas City, MO 64153
ATTN: Checktest/Service

c. Repair of Other NIRT Equipment. Official Inspection Agency managers must employ only qualified technicians to perform repairs on NIRT instruments used for official testing. Operators must notify the field office NIRT coordinator and the BAR when instruments malfunction. If the BAR determines the repair is major, the instrument monochromator must be checked before it is placed back into official service.

- d. NIRT Lamp Replacement. Official agencies may contact the BAR for Infratec model 1225/1226 replacement lamps. The defective lamp with lampholder must be sent to the BAR in order to obtain a replacement lamp. Fees will be assessed based on the hourly rate for repair, plus parts, and handling cost. Under normal circumstances, the total cost will not exceed \$100.00.

If the replacement lamp does not work upon receipt, or if it does work but the Infratec instrument displays an error code (e.g., Error 56, "No light is reaching the detector") contact the BAR immediately.

- e. Equipment Maintenance Log. Information entered into the log is used as a troubleshooting aid for repair personnel and provides the agency with a maintenance history of the instrument. Record any information pertaining to instrument repairs (e.g., lamp replacement) and other relevant information concerning unusual instrument operation.

CHAPTER 3

DETERMINING INSTRUMENT ACCURACY

Section Number	Section	Page Number
3.1	STANDARD REFERENCE AND SLOPE SAMPLE SETS	3-1
3.2	WHEAT BIAS ADJUSTMENTS	3-6
3.3	SOYBEAN BIAS ADJUSTMENTS	3-12
3.4	CORN BIAS ADJUSTMENTS	3-17
3.5	INSTRUMENT CHECKOUT	3-23

Attachment 1 - NIRT Daily Wheat SRS Worksheet

Attachment 2 - NIRT Daily Soybean SRS Worksheet

Attachment 3 - NIRT Weekly Wheat SRS Worksheet

Attachment 4 - NIRT Weekly Soybean SRS Worksheet

Attachment 5 - NIRT Wheat SRS Bias Log

Attachment 6 - NIRT Soybean SRS Bias Log

Attachment 7 - NIRT Daily Corn SRS Worksheet

Attachment 8 - NIRT Weekly Corn SRS Worksheet

Attachment 9 - NIRT Corn SRS Bias Log

CHAPTER 3

DETERMINING INSTRUMENT ACCURACY

3.1 STANDARD REFERENCE AND SLOPE SAMPLES

a. General.

- (1) Standard Reference (SRS) samples consist of bulk quantities of wheat, soybeans, and corn with established protein, oil, and starch values (as applicable) determined by ISE.
- (2) Slope samples are used to adjust the slope of the TSD NIRT instruments to agree optimally with the standard reference methods for each calibration. The average slope and intercept value from the TSD instruments are then used to determine the standard slope settings for each calibration.
- (3) SRS baseline values are corrected to a 12 percent moisture basis (mb) for wheat, 13 percent mb for soybeans, and a dry matter basis for corn before being released with the SRS sets.
- (4) SRS sets are used to test instrument accuracy and to determine the amount of bias adjustments needed. SRS may no longer be used if they weigh less than 500 grams.
- (5) Contact your NIRT coordinator or ISE to request SRS sample sets. The ISE will supply SRS that typically weigh between 650 and 750 grams for wheat, between 550 and 650 grams for soybeans, and between 575 and 675 grams for corn.

- b. Nature and Number of Samples. ISE will select a set of four (4) samples for Corn, and five (5) samples each of Hard Red Winter wheat (HRW), Hard Red Spring wheat (HRS), Soft Red Winter wheat (SRW), Soft White wheat (SWH), Hard White wheat (HDWH), Durum wheat (DU), and Soybeans. ISE will issue a unique baseline value for each class of wheat, for soybean protein and oil, and for corn protein, oil, and starch. Use the proper SRS set and baseline value for the class of wheat being tested. **Perform a duplicate determination on each wheat and corn SRS sample and a single determination on each soybean SRS sample.**

- c. Developing Baseline Values. The wheat protein, soybean protein and oil, and corn protein, oil, and starch baseline values are determined by ISE on the standard NIRT instrument. ISE instruments are directly compared and adjusted to optimally agree with the standard reference method.
- d. Replacing Reference Samples. ISE will maintain bulk reference samples to replace the field supply as they become infested, contaminated, or depleted. Testing locations must replace the wheat SRS set(s) after 6 months. The 6-month period starts on the day the SRS are first used. It should be within 2 weeks of receiving the SRS from ISE; if not, the SRS should be refrigerated until they are ready for use. Soybean and corn SRS sets will be replaced on a yearly basis unless they become infested, contaminated, or depleted. Request a replacement supply from ISE as far in advance as possible (30 days recommended).

Immediately notify your NIRT coordinator or ISE if any of the SRS sample(s) becomes contaminated or infested and obtain replacement(s).

In case of emergencies, SRS can be shipped overnight during the week. Otherwise, the SRS will be shipped through United Parcel Service (UPS) and should arrive within 5 business days of placing an order for replacement samples.

- (1) Collecting and Testing Replacement Samples. ISE will collect, evaluate, and select SRS. ISE will evaluate and select slope sets when calibrations are updated. ISE may request selected testing locations to provide samples for this purpose.
- (2) Replacing SRS or Baseline Values. A smooth transition should occur when new reference sample(s) and/or baseline value(s) are implemented. **This procedure does not apply to any changes associated with implementing a new calibration or standard slope setting.** Before replacing old reference sample set(s) or baseline value(s), perform the following:
 - (a) Check the instrument bias to the **old** baseline values using the old SRS. Record the average difference between the NIRT and baseline.
 - (b) Check, but do not correct, the instrument's bias term using the new SRS samples and baseline values. Record the average difference between the NIRT and baseline.
 - (c) Repeat steps (a) and (b) two additional times within a period not to exceed 2 weeks.

- (d) Review the three data sets. The new reference sample(s) with the new baseline value(s) may be used and the old reference sample(s) with the old baseline value(s) discarded if the amount of bias shown by the new set agrees with the amount of bias shown by the old set within an average of ± 0.05 percent protein or oil, or ± 0.20 starch.

Contact your NIRT coordinator or ISE for assistance if the new reference sample(s) do not meet the above requirements. Continue to use the old reference sample(s) for bias adjusting the instrument until the reason for the discrepancy is determined and corrected. If the new reference sample(s) meet the above requirement, they may now be used to check the instrument bias prior to running market samples.

- (3) Replacing SRS or Baseline Value(s) as a Result of Calibration Update or Adjustment. When a calibration update or adjustment and a new baseline are implemented simultaneously, no phase-in is required. Once the standard slope settings are entered in the instrument or on the calibration disk, test the new SRS two times. Use the Level II tolerances to determine if a bias adjustment is needed. Contact ISE with your final intercept values for the updated and/or adjusted calibrations.
- (4) Storage of Standard Reference Samples.
 - (a) Store reference samples at room temperature in plastic containers with screw tops or securely fastened lids and place away from vents, heating devices, and direct sunlight. To prevent insect infestation, operators may place mothballs sealed in plastic bags (with small holes) with the reference samples. The mothballs must remain sealed in the plastic bags at all times. Operators must remove any mothballs before testing the SRS set.
 - (b) SRS samples may be stored under refrigeration. However, the SRS must be allowed to reach room temperature ($\pm 5^{\circ}\text{F}$) before testing.
 - (c) Discard any SRS sample if it becomes infested or contaminated. Immediately inform your NIRT coordinator or ISE and obtain replacement sample(s).

- (d) Protect reference samples from manipulation by unauthorized persons to maintain sample integrity.
- e. Using Standard Reference Samples. Before testing market samples, the operator must first test, evaluate, and, if necessary, bias adjust the instrument using the appropriate SRS set and baseline values for soybeans, corn, or for the class of wheat being tested as follows:
- (1) Mix each SRS several times before testing.
 - (2) Analyze each sample according to the official procedures (see section 4.3.a).
 - (3) Review the NIRT values and apply the appropriate individual, average, and range differences between the NIRT and baseline value(s) (see section 3.1.f for wheat, 3.1.g for soybeans, and 3.1.h for corn).
 - (4) Record all data. Examples of a daily SRS worksheet, a weekly SRS worksheet, and a bias log are given in attachments 1-9. Testing locations may use these attachments or develop a log or worksheet to best suit their needs.
- f. Wheat Data Evaluation. Evaluate the SRS values by comparing individual and average NIRT protein to the corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein testing service:
- (1) Duplicate Difference. If the difference between the first and second analysis of an individual SRS exceeds ± 0.20 percent protein, then reanalyze the sample. Record the results from the two analyses closest to each other and discard the third result.

Duplicate Difference = an individual sample NIRT value first analysis minus (-) second individual sample NIRT value.
 - (2) Individual Difference. An individual reference sample NIRT protein value differs from its baseline value by **more than** ± 0.40 percent protein.

The individual difference is calculated as follows:

Individual difference = an individual sample NIRT protein value minus (-) sample baseline value.

- (3) Average Differences. The average difference between the SRS NIRT protein values and the baseline values exceeds the **applicable tolerance level (see section 3.2 for more information)**.
- g. Soybean Data Evaluation. Evaluate the SRS values by comparing individual and average NIRT protein and oil to their corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein and oil testing services:
- (1) Individual Difference. An individual reference sample NIRT protein value differs from its baseline value by **more than ± 0.40** percent protein. An individual reference sample NIRT oil value differs from its baseline value by **more than ± 0.30** percent oil.
- (2) Average Differences. The average difference between the SRS NIRT protein and/or oil values and the baseline values exceeds the **applicable tolerance level (see section 3.3 for more information)**.
- h. Corn Data Evaluation. Evaluate the SRS values by comparing individual and average NIRT protein, oil, and starch to their corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein, oil, and starch testing services:
- (1) Duplicate Difference. If the difference between the first and second analysis of an individual SRS exceeds ± 0.30 percent protein, ± 0.40 percent oil, or ± 0.90 percent starch, then reanalyze the sample for the constituent of interest. Record the results from the two analyses closest to each other for the constituent of interest and discard the third result.
- Duplicate Difference = an individual sample NIRT value first analysis minus (-) second individual sample NIRT value.
- (2) Individual Difference. An individual reference sample NIRT protein value differs from its baseline value by **more than ± 0.40** percent protein. An individual reference sample NIRT oil value differs from its baseline value by **more than ± 0.50** percent oil. An individual reference sample NIRT starch value differs from its baseline value by **more than ± 0.80** percent starch.

- (3) Average Differences. The average difference between the SRS NIRT protein and/or oil and/or starch values and the baseline values exceeds the **applicable tolerance level (see section 3.4 for more information)**.

3.2 WHEAT BIAS ADJUSTMENTS

a. General.

- (1) The NIRT instrument may, at times, either read high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official wheat protein testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to ISE standard NIRT instruments. Every effort shall be made to keep the calculated average bias of all NIRT instruments within ± 0.05 percent protein at all times. Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the calibration. This may mean running extra sets of SRS to have two or three valid sets available.
- (3) Prior to official wheat protein testing, perform a bias check and, if necessary, adjust the intercept constant for each wheat class being tested.
- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
- (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain bias records at each location at all times.

- b. Tolerance Levels. In addition to the individual sample difference limit of ± 0.40 percent protein, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available.

Presently, the four tolerance levels are set as follows:

(1) **LEVEL-I (± 0.10 percent protein tolerance):**

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 10 results).

(2) **LEVEL-II (± 0.07 percent protein tolerance):**

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 20 results) collected within a 2-week period and within a 5°F temperature range.

(3) **LEVEL-III (± 0.05 percent protein tolerance):**

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 30 results) collected within a 2-week period and within a 5°F temperature range.

(4) **LEVEL-IV (± 0.03 percent protein tolerance):**

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 50 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive or negative.

Record all bias adjustment data in the maintenance log and on the SRS log and/or worksheets (see attachments for examples). Include the date of the bias adjustment, the suspected source of error (if known), any action taken to correct the problem, direction provided by the NIRT coordinator or ISE, and the magnitude of the adjustment. All locations must submit a copy of all SRS information recorded during the week to the monitoring field office. The field office manager may request SRS information more frequently if a problem is suspected.

- c. Performing a Bias Check or Adjustment. Previous SRS NIRT results are invalidated after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than $\pm 5^{\circ}\text{F}$.

Perform a bias check: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instruments accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than $\pm 5^{\circ}\text{F}$ from the temperature recorded during the daily check.

Note: After changing between the 18 millimeter and 30 millimeter sample cell, select the high and low protein samples from one of the wheat SRS. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

STEP 1: Mix each SRS thoroughly before analyzing.

STEP 2: Calculate the difference between the duplicate analyses for the same sample. Does the duplicate difference for any sample differ by more than ± 0.20 percent protein?

- a. If NO, proceed to STEP 3.
- b. If YES, reanalyze the sample. Record and use the results from the two analyses closest to each other and discard the third result. Proceed to STEP 3.

STEP 3: Calculate individual analysis differences between the NIRT and baseline. Does any SRS differ by more than ± 0.40 percent protein from its baseline value?

- a. If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- b. If **YES**, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.50 percent protein?

The range difference is found by identifying the most positive individual difference and identifying the most negative individual difference then calculating the difference between the two extreme values. See the following examples for more information:

Example 1. Where positive and negative differences are observed:

If the largest positive difference is + 0.34 and the largest negative difference is - 0.39, then the range difference is $= (+ 0.34) - (- 0.39) = + 0.73$ percent protein.

Example 2. Where all observed differences are positive:

If the largest positive difference is + 0.34 and the smallest positive difference is +0.09, then the range difference is $= (+ 0.34) - (+ 0.09) = + 0.25$ percent protein.

Example 3. Where all observed differences are negative:

If the smallest negative difference is - 0.09 and the largest negative difference is -0.39, then the range difference is $= (- 0.09) - (- 0.39) = + 0.30$ percent protein.

- (1) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more ± 0.40 percent protein different from its baseline value and notify your NIRT coordinator. Calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- (2) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.

STEP 4: Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).

- a. Determine whether the average difference between the NIRT and baseline is ± 0.10 percent protein.
 - (1) If the average difference between the NIRT and baseline is ± 0.10 percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.b.
 - (2) If the average difference between the NIRT and baseline is greater than ± 0.10 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.05 percent protein or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- b. If the previous data set is valid, calculate the average difference from the baseline for the two sets (20 individual analyses).
 - (1) If the average difference is ± 0.07 percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.c.
 - (2) If the average difference is greater than ± 0.07 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.05 percent protein or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- c. If the previous three data sets are valid, calculate the average difference from the baseline for the three sets (30 individual analyses).

- (1) If the average difference is ± 0.05 percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.d.
 - (2) If the average difference is greater than ± 0.05 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.05 percent protein or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- d. If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (50 individual analyses), otherwise proceed to analyze market samples.
- (1) If the average difference is ± 0.03 percent protein or less, proceed to analyze market samples.
 - (2) If the average difference is greater than ± 0.03 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.05 percent protein or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.

3.3 SOYBEAN BIAS ADJUSTMENTS

a. General.

- (1) The NIRT instrument may, at times, read either high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official soybean protein and oil testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to ISE standard NIRT instruments. Every effort should be made to keep the calculated average bias of all NIRT instruments within ± 0.08 percent protein and ± 0.05 percent oil at all times.

Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the constituent of interest. This may mean running extra sets of SRS to have two or three valid sets available.

- (3) Prior to official soybean protein and oil testing, perform a bias check and, if necessary, adjust the intercept constant for each constituent.
- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
- (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain bias records at each location at all times.

- b. Tolerance Levels. In addition to the individual sample difference limit of ± 0.40 percent protein and ± 0.30 percent oil, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available.

Presently, the four tolerance levels are set as follows:

- (1) **LEVEL-I (± 0.17 percent protein and ± 0.12 percent oil tolerances):**

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 5 results).

- (2) **LEVEL-II (± 0.12 percent protein and ± 0.09 tolerances):**

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 10 results) collected within a 2-week period and within a 5°F temperature range.

- (3) **LEVEL-III (± 0.10 percent protein and ± 0.07 percent oil tolerances):**

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 15 results) collected within a 2-week period and within a 5°F temperature range.

- (4) **LEVEL-IV (± 0.08 percent protein and ± 0.05 percent oil tolerances):**

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 25 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive or negative.

- c. Performing a Bias Check or Adjustment. Previous SRS NIRT results are invalidated after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than $\pm 5^{\circ}\text{F}$.

Perform a bias check when: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instrument's accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than $\pm 5^{\circ}\text{F}$ from the temperature recorded during the daily check.

Note: After changing between the 18 millimeter and 30 millimeter sample cell, select the high and low protein SRS. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield protein results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

STEP 1: Mix each SRS thoroughly before analyzing.

STEP 2: Calculate individual sample differences between the NIRT and baseline. Do any SRS differ by more than ± 0.40 percent protein or ± 0.30 percent oil from its baseline value?

- a. If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.
- b. If **YES**, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.60 percent protein or 0.45 percent oil?

Examples for calculating the range difference between the two extreme values can be found on page 3-8.

- (1) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more ± 0.40 percent protein or ± 0.30 percent oil different from its baseline value and notify your NIRT coordinator. Only one sample can be dropped from the average. If more than one sample exceeds the tolerance, contact ISE.

Calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.

- (2) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.

STEP 3: Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).

- a. Determine whether the average difference between the NIRT and baseline is ± 0.17 percent protein or ± 0.12 percent oil.
 - (1) If the average difference between the NIRT and baseline is ± 0.17 percent protein or less and ± 0.12 percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 3.b.
 - (2) If the average difference between the NIRT and baseline is greater than ± 0.17 percent protein or ± 0.12 percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.08 percent protein or less or ± 0.05 percent oil or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.08 percent protein or ± 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- b. If the previous data set is valid, calculate the average difference from the baseline for the two sets (10 individual analyses).
 - (1) If the average difference is ± 0.12 percent protein or less and ± 0.09 percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 3.c.

- (2) If the average difference is greater than ± 0.12 percent protein or ± 0.09 percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.08 percent protein or less or ± 0.05 percent oil or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.08 percent protein or ± 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- c. If the previous three data sets are valid, calculate the average difference from the baseline for the three sets (15 individual analyses).
 - (1) If the average difference is ± 0.10 percent protein or less and ± 0.07 percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 3.d.
 - (2) If the average difference is greater than ± 0.10 percent protein or ± 0.07 percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.08 percent protein or less or ± 0.05 percent oil or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.08 percent protein or ± 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- d. If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (25 individual analyses) otherwise proceed to analyze market samples.
 - (1) If the average difference is ± 0.08 percent protein or less and ± 0.05 percent oil or less, proceed to analyze market samples.

- (2) If the average difference is greater than ± 0.08 percent protein or ± 0.05 percent oil or less, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.08 percent protein or less or ± 0.05 percent oil or less, proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.08 percent protein or ± 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.

3.4 CORN BIAS ADJUSTMENTS

a. General.

- (1) The NIRT instrument may, at times, read either high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official corn protein, oil, and starch testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to ISE standard NIRT instruments. Every effort should be made to keep the calculated average bias of all NIRT instruments within ± 0.05 percent protein, ± 0.06 percent oil, and ± 0.15 percent starch at all times.

Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the constituent of interest. This may mean running extra sets of SRS to have two or three valid sets available.

- (3) Prior to official corn protein, oil, and starch testing, perform a bias check and, if necessary, adjust the intercept constant for each constituent.

- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
 - (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
 - (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
 - (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
 - (8) Maintain bias records at each location at all times.
- b. Tolerance Levels. In addition to the individual sample difference limit of ± 0.40 percent protein, ± 0.50 percent oil, and ± 0.80 percent starch, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available.

Presently, the four tolerance levels are set as follows:

- (1) **LEVEL-I (± 0.15 percent protein, ± 0.15 percent oil, and ± 0.35 percent starch tolerances):**

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 8 results).

- (2) **LEVEL-II (± 0.10 percent protein, ± 0.10 percent oil, and ± 0.25 percent starch tolerances):**

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 16 results) collected within a 2-week period and within a 5°F temperature range.

- (3) **LEVEL-III (± 0.07 percent protein, ± 0.07 percent oil, and ± 0.20 percent starch tolerances):**

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 24 results) collected within a 2-week period and within a 5°F temperature range.

(4) **LEVEL-IV (± 0.05 percent protein, ± 0.06 percent oil, and ± 0.15 percent starch tolerances):**

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 40 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive nor negative.

- c. Performing a Bias Check or Adjustment. Previous SRS NIRT results are invalidated after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than $\pm 5^\circ\text{F}$.

Perform a bias check when: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instrument's accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than $\pm 5^\circ\text{F}$ from the temperature recorded during the daily check.

Note: After changing between the 18 millimeter and 30 millimeter sample cell, select the high and low oil SRS samples. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield oil results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

STEP 1: Mix each SRS thoroughly before analyzing.

- STEP 2:** Calculate the difference between the duplicate analyses for the same sample. Does the duplicate difference for any sample differ by more than ± 0.30 percent protein, or ± 0.40 percent oil, or ± 0.90 percent starch?
- a. If **NO**, proceed to STEP 3.
 - b. If **YES**, reanalyze the sample. Record and use the results from the two analyses closest to each other for the constituent of interest and discard the third result. Proceed to STEP 3.

- STEP 3:** Calculate individual analysis differences between the NIRT and baseline for each constituent. Does any SRS differ by more than ± 0.40 percent protein, or ± 0.50 percent oil, or ± 0.80 percent starch from its baseline value?
- a. If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
 - b. If **YES**, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.50 percent protein, or 0.60 percent oil, or 1.50 percent starch?

Examples for calculating the range difference between two extreme values can be found on page 3-9.

- (1) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more ± 0.40 percent protein, or ± 0.50 percent oil, or ± 0.80 percent starch different from its baseline value and notify your NIRT coordinator. Calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- (2) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.

- STEP 4:** Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).
- a. Determine whether the average difference between the NIRT and baseline is ± 0.15 percent protein, or ± 0.15 percent oil, or ± 0.35 percent starch.

- (1) If the average difference between the NIRT and baseline is ± 0.15 percent protein or less, or ± 0.15 percent oil or less, or ± 0.35 percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.b.
 - (2) If the average difference between the NIRT and baseline is greater than ± 0.15 percent protein, or ± 0.15 percent oil, or ± 0.35 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.07 percent protein or less, or ± 0.07 percent oil or less, or ± 0.20 percent starch or less proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.07 percent protein, or ± 0.07 percent oil, or ± 0.20 percent starch, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- b. If the previous data set is valid, calculate the average difference from the baseline for the two sets (16 individual analyses).
- (1) If the average difference is ± 0.10 percent protein or less, or ± 0.10 percent oil or less, or ± 0.25 percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.c.
 - (2) If the average difference is greater than ± 0.10 percent protein, or ± 0.10 percent oil, or ± 0.25 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.07 percent protein or less, or ± 0.07 percent oil, or ± 0.20 percent starch proceed to analyze market samples.

- (b) If the difference is still greater than ± 0.07 percent protein, or ± 0.07 percent oil, or ± 0.20 percent starch recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- c. If the previous three data sets are valid, calculate the average difference from the baseline for the three sets (24 individual analyses).
 - (1) If the average difference is ± 0.07 percent protein or less, or ± 0.07 percent oil or less, or ± 0.20 percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to step 4.d.
 - (2) If the average difference is greater than ± 0.07 percent protein, or ± 0.07 percent oil, or ± 0.20 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - (a) If the corrected difference is ± 0.07 percent protein or less, or ± 0.07 percent oil or less, or ± 0.20 percent starch or less proceed to analyze market samples.
 - (b) If the difference is still greater than ± 0.07 percent protein, or ± 0.07 percent oil, or ± 0.20 percent starch recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- d. If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (40 individual analyses) otherwise proceed to analyze market samples.
 - (1) If the average difference is ± 0.05 percent protein or less, or ± 0.06 percent oil or less, or ± 0.15 percent starch or less proceed to analyze market samples.
 - (2) If the average difference is greater than ± 0.05 percent protein, or ± 0.06 percent oil, or ± 0.15 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.

- (a) If the corrected difference is ± 0.07 percent protein or less, or ± 0.07 percent oil or less, or ± 0.20 percent starch or less proceed to analyze market samples.
- (b) If the difference is still greater than ± 0.07 percent protein, or ± 0.07 percent oil, or ± 0.20 percent starch recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.

3.5 INSTRUMENT CHECKOUT

Certain checks and maintenance steps must be performed to verify that the NIRT instruments are functioning properly prior to providing official testing services.

a. Instrument Checkout Schedule.

- (1) Locations providing NIRT testing on a daily basis must complete these checks for each day.
- (2) Locations providing infrequent NIRT testing and/or those that monitor NIRT activities of specified service points must complete these checks before any official NIRT results are provided.
- (3) The instrument checkout procedures outlined below are general. If the instrument does not pass the checkout sequence, official testing shall be suspended until the problem is resolved or corrected. **The first step toward resolution is to repeat the test in question and seek advice from your NIRT coordinator. If the problem cannot be resolved, seek advice from ISE.**

b. Instrument Checkout Procedures.

- (1) Each instrument must have the monochromator checked, the sample cell pathlength standardized (unless you have an Infratec 1229 with a variable sample cell), and use the appropriate standard slope settings for each class of wheat tested and/or soybeans and/or corn tested at the specified service point.

- (2) Analyze the appropriate SRS following official procedures each day or prior to use, as applicable (see 3.5.a).
 - (3) Dust out the sample hopper and path at the end of each day.
 - (4) If more than one instrument is being used at a location, check to make sure the correct calibration disk is being used. Each instrument has its own specific calibration disk labeled with the unit's serial number and the instrument's "O" and "P" constants entered on the disk.
- c. Other Tests. The NIRT instrument performs extensive operational checks on itself whenever it is powered on and during operation. Record any error messages that appear for use in troubleshooting instrument problems.

It is the responsibility of the NIRT coordinator and technicians to alert the supervisor and suspend official testing if the NIRT performance is questionable. **Use the SRS to test instrument accuracy if instrument performance is questioned.** Official testing may resume when acceptable instrument performance is demonstrated.

NIRT Daily Wheat SRS Worksheet

Location: _____ Serial #: _____ Date: _____
 Operator: _____ Temperature: _____ R.Humidity: _____
 Instrument Constants: "O" _____ "P" _____
 Protein Constants: Slope _____ Intercept _____
 Wheat Class _____

PROTEIN					
SRS	Value	Run 1	Difference	Repeat	Difference
1					
1					
2					
2					
3					
3					
4					
4					
5					
5					
Average					

Range of Differences (Maximum 0.50)

Bias Calculation:

Protein Average
Minus Baseline
Today's Bias

Transfer range and bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust the intercept constant for a constituent if it is out of tolerance.

REPEAT RULES:

1. Repeat any sample if an outlier is reported.
2. Repeat an individual SRS if the difference between the first and second analysis exceeds 0.20 percent. Record the results from the two analyses closest to each other and discard the third result.
3. Repeat any individual SRS that deviates by more than 0.40 from its target value and the range tolerances are exceeded.
4. If an individual SRS deviates from its target by more than 0.30 for five consecutive runs, contact the BAR to have the SRS replaced.

NIRT Daily Soybean SRS Worksheet

Location: _____ Serial #: _____ Date: _____
 Operator: _____ Temperature: _____ R.Humidity: _____
 Instrument Constants: "O" _____ "P" _____
 Protein Constants: Slope _____ Intercept _____
 Oil Constants: Slope _____ Intercept _____

PROTEIN					
SRS	Value	Run 1	Difference	Repeat	Difference
1					
2					
3					
4					
5					
Average					

Range of Differences (Maximum 0.60)

OIL					
SRS	Value	Run 1	Difference	Repeat	Difference
1					
2					
3					
4					
5					
Average					

Range of Differences (Maximum 0.45)

Bias Calculation:

Protein Average

Minus Baseline

Today's Bias

Oil Average

Minus Baseline

Today's Bias

Transfer range and bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust the intercept constant for a constituent if it is out of tolerance.

REPEAT RULES:

1. Repeat any sample if an outlier is reported.
2. Repeat the SRS set if the range tolerances are exceeded.
3. If the range tolerances are exceeded on two consecutive runs and an individual SRS deviates by 0.40 for protein and 0.30 for oil, that sample may be temporarily dropped from the average. Contact the BAR for a replacement.

NIRT Weekly Wheat SRS Worksheet

Wheat Class _____

Location: _____

Instrument Constants:

Protein Constants:

Serial # _____

"O" _____

Slope _____

Date: _____

"P"

Intercept _____

Date:

Tech:

Temp:

Humidity:

[illegible]

Range of Differences (Maximum 0.50)

--	--	--	--	--

Bias Calculation:

One Run (.10)

Two Runs (.07)

Three Runs (.05)

Five Runs (all +/-)(.03)

2nd Previous Run

Previous Run

	2nd Previous Run								
	Previous Run								

NIRT Weekly Soybean SRS Worksheet

Location: _____
Instrument Constants: _____
Protein Constants: _____

Serial # _____
"O" _____
Slope _____

Date: _____
 "P" _____
 Intercept _____

Date
Tech
Temp
Humidity

[illegible]

Range of Differences (Maximum 0.60)

☐ ☐ ☐ ☐ ☐

Bias Calculation:

One Run (.17)

Two Runs (.12)

Three Runs (.10)

Five Runs (all +/-)(.08)

2nd Previous Run

Previous Run

	2nd Previous Run								
	Previous Run								

Constants:

Slope _____

Intercept _____

[illegible]

Range of Differences (Maximum 0.45)

--	--	--	--	--

Bias Calculation:

One Run (.12)

Two Runs (.09)

Three Runs (.07)

Five Runs (all +/-)(.05)

2nd Previous Run

Previous Run

	2nd Previous Run								
	Previous Run								

NIRT Wheat SRS Bias Log

Tecator Model: _____ Serial #: _____ Location: _____

NOTE: Only results less than two weeks old and within a 5 °F temperature range may be averaged. Results obtained after a slope or bias adjustment, SRS replacement, or NIRT repair or replacement cannot be averaged with results obtained prior to the change (also, the 5-Run correction can only be applied if five consecutive, valid runs are all positive or all negative). Every effort should be made to maintain the bias within the 0.05% Protein tolerance.

[illegible]

*(S) slope adjustment, (B) bias adjustment, or (R) SRS replaced. If the NIRT is repaired or replaced, start a new log.

NIRT Daily Corn SRS Worksheet

Location: _____ Serial #: _____ Date: _____
Operator: _____ Temperature: _____ R. Humidity _____
Instrument Constants: "O" _____ "P" _____

Protein Oil Starch
Slope _____
Intercept _____

S R S	Protein					Oil					Starch				
	Value	Run 1	Diff	Repeat	Diff	Value	Run 1	Diff	Repeat	Diff	Value	Run 1	Diff	Repeat	Diff
1															
1															
2															
2															
3															
3															
4															
4															
A v g															

Range of Differences _____
Maximum Limit 0.50 0.60 1.50

Level I Tolerances (average of 8 results): Protein = ± 0.15 Oil = ± 0.15 Starch = ± 0.35

Transfer bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust intercept constant for a constituent if it is out of tolerance.

Weekly NIRT Corn SRS Worksheet

Constituent: _____ Location: _____ Serial #: _____
Date: _____ Operator: _____
Temperature: _____ R. Humidity: _____

Instrument Constants: "O": _____ "P": _____
Slope: _____ Intercept: _____

Date:

Tech:

Temp:

RH:

SRS	Baseline	Result	Diff	Result	Diff	Result	Diff	Result	Diff	Result	Diff
1											
1											
2											
2											
3											
3											
4											
4											
Avg											
Range											

Transfer bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust intercept constant for a constituent if it is out of tolerance.

CHAPTER 4

SAMPLE PREPARATION AND ANALYSIS

Section Number	Section	Page Number
4.1	BASIS OF DETERMINATION	4-1
4.2	CLEANING SAMPLES	4-2
4.3	NIRT ANALYSIS	4-3
4.4	OUTLIER INDICATIONS	4-4
4.5	TESTING INDIVIDUAL AND SUBLOT SAMPLES	4-6
4.6	CERTIFYING OFFICIAL RESULTS	4-6
4.7	CONVERTING RESULTS TO AN ALTERNATE MOISTURE BASIS	4-7

CHAPTER 4

SAMPLE PREPARATION AND ANALYSIS

4.1 BASIS OF DETERMINATION

NIRT wheat protein is determined on a representative sample portion of approximately 500 grams up to the work portion size of wheat after the removal of dockage using a standard dockage tester. NIRT soybean protein and oil are determined on a representative portion of approximately 500 grams up to the work portion size of cleaned soybeans. NIRT corn protein, oil, and starch are determined on a representative portion of approximately 500 grams up to the work portion size after the removal of broken corn and foreign material. An NIRT instrument equipped with a flow through sample cell is approved equipment for official testing. An NIRT instrument equipped with the sample transport mechanism can be approved (ISE will approve these instruments on a case-by-case basis) for use in analyzing an approximately 80-gram wheat sample and 175-gram samples of corn and soybeans. Contact ISE for more information on the use of the sample transport mechanism.

The official NIRT calibrations were developed to automatically provide protein percentages corrected to a 12 percent moisture basis for wheat, protein and oil in soybeans to a 13 percent moisture basis, and protein, oil, and starch in corn on a dry matter basis. This eliminated the need for mathematical calculation.

Since ISE provides separate calibrations for each wheat class, it is essential that the correct calibration is used. A qualified inspector must review the wheat sample to determine class to ensure that the appropriate calibration is used. Use the calibration for the predominant class when performing an NIRT protein determination on a mixed wheat sample.

As the sample passes through the light path, multiple measurements of the transmitted light takes place. Each time a measurement is taken, the portion of the sample within the light path is considered a subsample for NIRT purposes.

4.2 CLEANING SAMPLES

a. Wheat.

Samples shall be cleaned by a mechanical process (dockage tester) that removes dockage. The dockage tester uses aspiration (air) and a combination of riddles and sieves to remove any readily separable foreign matter. Generally, the foreign matter removed consists of all matter lighter, larger, or smaller than grain. If excessive quantities of wild buckwheat, cob joints, chaff and similar types of seeds, and flaxseed are found additional sieving procedures are required. Refer to the Wheat Chapter in Book II of the Grain Inspection Handbook for detailed instructions.

b. Soybeans.

Samples shall be cleaned by hand sieving over an 8/64-inch round hole sieve to remove any fine foreign material (FM). **The portion remaining on top of the sieve shall be handpicked to remove any coarse FM (refer to chapter 1, section 1.3 for the definition of coarse FM).** The portion remaining on top of the sieve after the removal of fine and coarse FM shall be used for the analysis.

c. Corn.

Broken corn and foreign material (BCFM) shall be removed by sieving the sample (machine sieving with a dockage tester or hand sieving) over an 12/64-inch round hole sieve **and handpicking any material (e.g., soybeans, pieces of corn cob) other than corn that remains on top of the sieve.** The portion remaining on top of the sieve after the removal of BCFM shall be used for the analysis.

d. Mechanical/Hand Sieving Procedures.

Use the following procedures to mechanically/hand sieve foreign material (FM in soybeans, BCFM in corn).

(1) For mechanical sieving:

- (a) Place the appropriate sieve on top of a bottom pan and position it in the mechanical grain sizer.
- (b) Pour the sample portion into the center of the sieve. (Do not sieve more than 500 grams at a time.)
- (c) Set the sizer's counter to the appropriate number of strokes (20 for corn, and 5 for soybeans) and then turn it on.

- (2) For hand sieving:
 - (a) Place the appropriate sieve on top of a bottom pan.
 - (b) Pour the sample portion into the center of the sieve. (Do not sieve more than 500 grams at a time.)
 - (c) Hold the sieve and bottom pan level and, using a steady motion, move the sieve from right to left approximately 10 inches. Return from left to right to complete one sieving operation. Repeat this operation 20 times for corn, and 5 times for soybeans.

4.3 NIRT ANALYSIS

Operators must read the user's manual and familiarize themselves with the instructions before operating the NIRT instrument.

- a. Locations Using the Flow Through Sample Cell.
 - (1) For wheat protein determinations, use the 18-millimeter cell in the NIRT analyzer. The protein portion shall be a representative amount (dockage-free), weighing between 500 grams and the work portion size. For soybean protein and/or oil determinations, use the 30-millimeter cell in the NIRT analyzer. The analytical portion shall be a representative amount weighing between 500 grams and the work portion size that has been cleaned using the procedures found in section 4.2 of this chapter. For corn protein, oil, and/or starch determinations, use the 30-millimeter cell in the NIRT analyzer. The analytical portion shall be a representative amount (broken corn and foreign material free) weighing between 500 grams and the work portion size. **The operator must ensure that the sample's temperature is between 60°F and 80°F (16°C and 27°C).** If the operator has a reason to suspect that the sample may not be within the acceptable temperature range, use a liquid-in-glass or digital thermometer specified to $\pm 2^\circ\text{F}$ ($\pm 1^\circ\text{C}$) accuracy or better to measure the sample temperature. If the sample is not in the acceptable temperature range, allow it to cool or warm to within the limits before testing.

- (2) Ensure that the displayed calibration ID is the ISE approved calibration for the type of grain and wheat class being tested and that the SRS for that grain type or wheat class have been tested that day.
 - (3) Pour the entire sample into the NIRT hopper, enter the sample identification number, and press the "RUN" button. The instrument provides a display (and printout, if a printer is attached) of percent protein for wheat, percent protein and oil for soybeans, and percent oil, protein, and starch for corn.
 - (4) Retain the sample for further analysis if needed.
- b. Locations Using a Sample Transport Mechanism. The sample transport mechanism is needed when samples do not meet the requirement for the flow through sample cell. Contact ISE for the sample transport mechanism procedures.

4.4 OUTLIER INDICATIONS

An outlier indication is a warning that the result may be in error. Outlier indications should rarely occur. If frequent outlier indications are observed, contact ISE.

- a. Types of Outliers. There are four possible outlier indicators (A, B, C, and D). The values for each indicator can range in degree from 0 (no outlier) to 5 (very extreme). The greater the value, the less reliable the predicted result. Review the user's manual for more information.
- (1) "A" Outlier (Residual). The sample's scan doesn't fit the calibration.
 - (2) "B" Outlier (Leverage). The sample's scan doesn't fit the calibration and, if added, will have a strong influence.
 - (3) "C" Outlier (Standard Deviation). The standard deviation (variability) between subsamples is beyond the specified limit.
 - (4) "D" Outlier (Outside Range Limits). One or more of the subsamples have been predicted above or below the constituent value limits for the calibration.
- b. Causes of Outliers. The most likely causes for outliers are:
- (1) Wrong calibration selected. Testing a wheat sample with the wrong wheat calibration or the soybean calibration;

- (2) Wrong sample cell. Testing a wheat sample using the soybean/corn (30 mm) cell or vice versa;
- (3) Extremely dirty samples;
- (4) Poorly mixed samples;
- (5) Sample packing or settling problems;
- (6) Electrical interference;
- (7) Measurement of samples not represented in the calibration set; or
- (8) Improper instrument warm up.

Some of these conditions will cause intermittent outlier indications (not repeated on reruns) and others will cause consistent outlier indications. In general, an outlier indication is a warning that the result may be in error.

- c. What to do when an outlier occurs? When any outlier indication occurs, the operator must perform the following:
 - (1) Verify that the correct calibration and sample cell was used (18 mm for wheat and 30 mm for soybeans and corn).
 - (2) Make sure the sample is cleaned per official procedures.
 - (3) Make sure the room and sample temperature and the room relative humidity are within the specified ranges.
 - (4) Mix the sample thoroughly before retesting.
 - (5) Rerun the sample through the Infratec.
 - (6) If the second run does not show an outlier indication, certify the results of the second run.

- (7) If the second run shows an outlier indication, certify on the basis of the average of the two runs.
- (8) Never certify a result with a level "5" outlier in the "A" or "B" positions.

NOTE: The NIRT determines both protein and oil in soybeans, and protein, oil, and starch in corn on each run. Use the second run result for the constituent in question only. Use the first run result for the other constituent.

4.5 TESTING INDIVIDUAL AND SUBLOT SAMPLES

- a. Individual Sample. Official sample-lot or submitted samples of wheat, corn, or soybeans are tested and certificated for protein, oil, or starch as applicable, in conjunction with official grade determinations or as a separate testing service.
- b. Shiplot and Lash Barges. Testing for protein in wheat is based on subplot results in accordance with the cu-sum loading plan. Testing for protein and/or oil in soybeans and for protein, and/or oil, and/or starch in corn can be determined on an average of individual subplot results or on the basis of a composite sample representing the entire lot. Refer to the Grain Inspection Handbook, Book III, for details concerning the cu-sum loading plan.
- c. Unit Trains. Testing for protein in wheat is determined on individual or subplot (up to five carriers per subplot) basis in accordance with the cu-sum loading plan. Testing for protein and/or oil in soybeans and for protein, and/or oil, and/or starch in corn can be determined on individual, subplot (up to five carriers per subplot) or composite sample basis. Applicants can request corn protein, oil, and/or starch, and soybean protein and/or oil on a subplot basis while requesting inspection for grade on an individual carrier basis. When articulated railcars are used, each car is tested as a subplot.

4.6 CERTIFYING OFFICIAL RESULTS

- a. Moisture Basis. NIRT instruments are programmed to determine official criteria results (i.e., protein, oil, starch) on a moisture basis that is commonly used for trading purposes. The instruments will automatically report wheat protein results on a 12.0 percent moisture basis, soybean protein and oil results on a 13.0 percent moisture basis, and corn protein, oil, and starch results on a dry matter basis.

- b. General Procedures. All official criteria results (e.g., protein, oil, starch) shall be recorded on the work record and in the "Remarks" section of the official inspection certificate to the nearest tenth percent on the applicable moisture basis (wheat 12.0 percent, soybeans 13.0 percent, corn - dry matter basis). Upon request, an applicant can request an alternate moisture basis be used for certifying results for soybean protein and oil, or corn protein, oil, and starch. Show the official results on the inspection certificate using an approved statement as shown in Grain Inspection Handbook, Book IV. Upon request of the applicant for service, official criteria results may be stated on letterhead stationery in lieu of an official certificate.

4.7 CONVERTING RESULTS TO AN ALTERNATE MOISTURE BASIS

- a. Conversion Formulas.

Examples of alternate moisture basis specifications, and formulas for correcting the NIRT results are:

- (1) Converting a soybean protein/oil, or corn protein/oil/starch result from the standard moisture basis to an "as is" moisture basis.

$$A = \frac{P \times (100 - M)}{C}$$

Where A = Percent protein/oil/starch on an "as is" moisture basis.

P = NIRT protein/oil/starch result on the standard moisture basis (based on NIRT result rounded to the nearest tenth percent).

M = Official moisture result for the sample/lot (as applicable).

C = 87 for soybeans, or 100 for corn

- (2) Converting a corn protein, oil, or starch result from the dry matter basis to another specified moisture basis.

$$A = \frac{P \times (100 - M)}{100}$$

Where A = Percent protein/oil/starch on a specified moisture basis.

P = NIRT protein/oil/starch result on a dry matter basis (based on NIRT result rounded to the nearest tenth percent).

M = Moisture basis specified by applicant.

- (3) Converting a soybean protein or oil result from the 13.0 percent moisture basis to another specified moisture basis.

$$A = \frac{P \times (100 - M)}{87}$$

Where A = Percent protein/oil on a specified moisture basis.

P = NIRT protein/oil result on the 13.0 percent moisture basis (based on NIRT result rounded to the nearest tenth percent).

M = Moisture basis specified by applicant.

- (4) Converting a soybean protein result to an oil-free and moisture-free basis.

$$B = \frac{P \times 100}{(100 - (O + 13))}$$

Where B = Percent protein on an oil-free and moisture-free basis.

P = NIRT protein result on a 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent).

O = Percent oil on a 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent).

- (5) Converting a soybean protein result to an oil-free and specified moisture basis.

$$C = \frac{P \times (100 - M)}{100 - (13 + O)}$$

Where C = Percent protein on an oil-free and specified moisture basis.

P = NIRT protein result on a 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent).

M = Moisture basis specified by the applicant.

O = NIRT oil result on 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent).

b. Conversion Guidelines.

- (1) On submitted samples and single lots, use the official moisture result for the lot if the applicant requests an "as is" moisture basis.
- (2) If subplot testing is requested for corn protein/oil/starch, or soybean protein/oil, on unit trains, lash barges, or ships inspected as single lots, and the applicant does not specify limits for protein/oil/starch content on an alternate moisture basis, record individual subplot results on the standard moisture basis. Upon completion of loading, convert the final average protein/oil/starch results to the specified moisture basis. For an "as is" moisture basis, use the official average moisture result in the conversion formula.
- (3) If a load order specifies limits on the protein/oil/starch content on an alternate moisture basis, convert the individual subplot protein/oil/starch results to the desired moisture basis. Use the subplot official moisture result if the applicant requested an "as is" moisture basis.

U.S. DEPARTMENT OF AGRICULTURE
GRAIN INSPECTION, PACKERS AND
STOCKYARDS ADMINISTRATION
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NIRT HANDBOOK
CHAPTER 5
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CHAPTER 5

NIRT MONITORING PROGRAM

Section Number	Section	Page Number
5.1	GENERAL INFORMATION	5-1
5.2	MONITORING REFERENCE SAMPLES	5-3
5.3	MONITORING WHEAT FILE SAMPLES	5-3
5.4	LOCAL NIRT MONITORING	5-5
5.5	NIRT CHECK SAMPLES	5-5
5.6	WHEAT QUALITY CONTROL CHARTS	5-5
5.7	COLLABORATIVE CHECK SAMPLES AND SPECIAL STUDIES	5-9
5.8	INTERMARKET SAMPLE EXCHANGE	5-9
	Figure 1 - Average Difference Between Field Location and ISE	5-11
	Figure 2 - Range Difference Between Field Location and ISE	5-12

CHAPTER 5

NIRT MONITORING PROGRAM

5.1 GENERAL INFORMATION

- a. Wheat Protein. The protein monitoring program is designed to monitor the accuracy of official wheat protein testing services. Protein accuracy is evaluated by comparing field office and specified service point results with ISE results. Field offices with NIRT instruments may conduct supplemental file sample monitoring of specified service points within the circuit in addition to ISE monitoring to measure the performance of instruments and technicians.

Monitoring information is used by specified service points to evaluate the performance of their protein testing program. Field offices use the monitoring information to evaluate their own protein accuracy as well as the accuracy of the protein testing service program within their circuit. ISE uses the monitoring information to evaluate the accuracy of individual field locations as well as the entire program.

ISE monitoring results will identify service points generating questionable protein results. Field offices and specified service points must initiate corrective action and follow up whenever wheat protein testing problems are detected. Corrective action and follow-up includes investigating, identifying, correcting, and documenting the cause of protein accuracy problems.

Several methods are utilized to monitor particular elements in the protein testing process. These methods include quality control charts for file samples monitored, prepared samples, intermarket sample exchanges, and special studies. Procedures regarding these monitoring methods are discussed in the following sections.

- b. Soybean Protein and Oil. The protein and oil check sample program is designed to monitor the accuracy of official soybean protein and oil testing services. Protein and oil accuracy is evaluated by comparing field office and specified service point results with ISE results.

Check sample information is used by field offices and inspection agencies to evaluate the performance of their local protein and oil testing program. ISE uses the check sample information to evaluate the accuracy of individual field locations as well as the entire program. Local monitoring by the field office will identify service points having questionable protein and/or oil results for all NIRT operators in their circuit.

Field offices and specified service points must initiate corrective action and follow-up whenever soybean protein and/or oil testing problems are detected. Corrective action and follow-up includes investigating, identifying, correcting, and documenting the cause of protein and/or oil accuracy problems.

Several methods are utilized to monitor particular elements in the protein and oil testing process. These methods include local monitoring, check samples, opinion samples, intermarket sample exchanges, and special studies or collaboratives. Procedures regarding these monitoring methods are discussed in the following sections.

- c. Corn Protein, Oil, and Starch. The protein, oil, and starch check sample program is designed to monitor the accuracy of official corn protein, oil, and starch testing services. Protein, oil, and starch accuracy is evaluated by comparing field office and specified service point results with ISE results.

Check sample information is used by field offices and inspection agencies to evaluate the performance of their local testing program. ISE uses the check sample information to evaluate the accuracy of individual field locations as well as the entire program.

Local monitoring by the field office will identify service points having questionable protein, oil, and/or starch results for all NIRT operators in their circuit. Field offices and specified service points must initiate corrective action and follow-up whenever corn protein, oil, and/or starch problems are detected. Corrective action and follow-up includes investigating, identifying, correcting, and documenting the cause of protein, oil, and/or starch accuracy problems.

Several methods are utilized to monitor particular elements in the protein, oil, and starch testing process. These methods include local monitoring, check samples, opinion samples, intermarket sample exchanges, and special studies or collaboratives. Procedures regarding these monitoring methods are discussed in the following sections.

5.2 MONITORING REFERENCE SAMPLES

Daily SRS results will be monitored to evaluate instrument accuracy, bias adjustment procedures, and technician consistency. In addition, this information will be used to determine sources of error and formulate corrective action. All locations must submit a copy of SRS information recorded for each weekly period to the monitoring field office. The field office manager may request SRS information more frequently if a problem is suspected.

- a. SRS Sample Monitoring Frequency. ISE, monitoring field offices, and specified service points record the results of daily and weekly standard reference sample checks on the standard reference sample form (examples are shown in Chapter 3). Specified service points and field office NIRT coordinators shall review these forms for accuracy and completeness.
- b. Evaluation of Results. NIRT coordinators will evaluate the SRS test results to determine if: (1) bias adjustments were completed when necessary, (2) instrument accuracy was maintained, or (3) bias adjustments were required frequently (which may indicate the need for operator training, instrument repair, or SRS replacement).

5.3 MONITORING WHEAT FILE SAMPLES

Wheat protein accuracy is evaluated through the official monitoring system. Official inspection points will select and forward samples to ISE. Protein accuracy is determined by comparing original protein results with the average of the ISE master instruments results.

Agencies and field offices will initiate follow-up action when protein testing deficiencies (absolute, tolerance, or run limit violations) are detected by the monitoring program. Official agencies shall forward samples selected for the field office's supplemental monitoring program to the designated field office location.

- a. Selecting Samples. Specified service points and field offices providing original protein testing services shall use the following procedures to select monitor samples. Select five wheat samples per class tested, per week, representing the range of protein observed during the week. Include a low and a high protein sample each week with three intermediate samples. Avoid selecting all samples tested from the same day. When less than five samples per class are tested during a week, select all samples tested for that class. Do not make up monitoring samples to fulfill the minimum number of samples. Do not select mixed wheat samples.

Each dockage free sample submitted for monitoring must be at least 500 grams unless special arrangements have been made with ISE.

Field offices and/or ISE may request additional samples for monitoring and/or special study purposes.

- b. Mailing Instructions. Seal the samples in individual 6-mil plastic bags. Mark the sample number and wheat class on each bag using an indelible marker. Place the samples and sample information (scan sheet) in a canvas mailing bag and indicate "Protein Monitoring Sample" on the reverse side of the mail tag (use an orange color mailing tag). This will assist in separating protein monitoring samples from other samples received in the mail. According to postal requirements, Federal employees must use metered mailing tags. All nonfederal employees (i.e., delegated and designated agencies) must use business reply mailing tags.
- c. Monitoring Results. Field office personnel must retrieve the monitoring data from the Quality Assurance/Quality Control (QAQC) homepage, review the information, and immediately initiate follow-up action when accuracy deficiencies are indicated by monitoring results. In addition, field office managers must forward ISE monitoring results to the specified service points for their review and follow-up action within 2 working days of retrieval.
- d. Evaluating Monitoring Results. Field offices and specified service points will evaluate completed quality control charts to determine if any action limit (tolerance limits, absolute limits, and/or run limit) violations occurred.

Action limit violations occurring on the average difference chart generally indicate a bias-related problem. Action limit violations occurring on the range difference chart generally indicate inconsistency due to fluctuating laboratory conditions, failure to follow procedures consistently, instrument problems, or improper instrument slope or bias adjustment. Violations on the range difference chart are actually more serious than those on the average difference chart because if the results are inconsistent, the average differences are not meaningful.

Monitoring field offices and agencies must initiate corrective action when quality control chart rule violations occur. Field office managers must document any action taken to resolve the differences. This documentation includes action taken to identify the cause and extent of the problem and steps to resolve the problem and/or reasons why no further action is necessary. Documentation may be placed directly on the control chart indicating action limit violations.

5.4 LOCAL NIRT MONITORING

Field offices may conduct periodic or routine file sample monitoring of official laboratories providing testing (soybean protein and oil, and/or corn protein, oil, and starch) in their circuit to measure the performance of instruments and technicians. ISE will advise the field on developing a local monitoring program and assist in analyzing and interpreting the data.

5.5 NIRT CHECK SAMPLES

Check samples are used to test the capability of the national soybean protein and oil, and the corn protein, oil, and starch testing programs. Soybean check samples are issued twice per year and corn check samples are issued once per year (prior to harvest) by ISE. Specified service points and laboratories that provide soybean protein and oil, and/or corn protein, oil, and starch testing services for FGIS must participate in the check sample program.

Check samples originate from ISE and will include specific instructions for testing the samples. Upon receipt, inspection points must complete the analysis of these samples within 10 working days. Report check sample results to ISE on the forms provided. Whenever possible, the results should be faxed to ISE to expedite the preparation of the check sample report.

The field will be notified immediately if the test results indicate a problem at a test location. Otherwise, ISE will not release any information on the content of the check samples until the results from all laboratories have been received. ISE will analyze the data and issue a report which will be distributed to all participating official agencies and supervising field offices.

When requested, field offices will assist ISE in obtaining bulk soybean or corn samples to be used in the check sample program.

5.6 WHEAT QUALITY CONTROL CHARTS

- a. General. A Quality Control Chart (QCC) is a visual display of monitoring data. A QCC effectively displays extreme variations, shifts, and trends. Also, a QCC illustrates the difference between results while statistically defining expected variability using control limits. These limits are established based on the normal expected variation of results.

- b. Control Charts. The protein monitoring program utilizes Average Difference and Range Difference quality control charts. The average difference chart illustrates the difference between a specified service point's average for five weekly monitoring samples and the ISE's average for the same samples. The range difference chart plots the range of individual sample differences for the corresponding weekly monitoring sample set.

- (1) Average Difference Chart. The average difference chart includes a zero or Center Line (CL), upper and lower Tolerance Limits (TL), and upper and lower Absolute Limit lines (AL).

The center line is the control chart reference point. Points plotted above the center line indicate a positive difference when compared to the monitoring result. Points plotted below the center line indicate a negative difference when compared to the monitoring result. The absolute limit lines are set at ± 0.20 percent protein from the center line. The tolerance limit lines are set at ± 0.15 percent protein from the center line.

These tolerances are determined statistically based on the systems actual performance and may be revised from time to time. A violation of any of the established tolerances means that there is less than one chance in one hundred that the observed error level occurred due to random chance. Therefore, it is very likely that a correctable problem exists.

- (2) Range Difference Chart. The range difference chart indicates how much difference is observed within a set of monitoring samples. The absolute limit line is set at 0.60 percent protein. The tolerance limit line is set at 0.40 percent protein.

Average and Range Difference control charts are shown in Figures 1 and 2.

- c. Plotting Control Charts. Control charts are generated as follows.

- (1) Sequential Plotting. Control charts depict how a process or system is varying over time. In order for the results to be meaningful, data is plotted in the sequence in which the original results were obtained. That is, when plotting the Average/Range chart, samples originally tested during week 3 are plotted after samples originally tested during week 2, but before samples originally tested during week 4 (see **Figures 1 and 2**).

- (2) Average Difference. The difference between the original average result and the monitoring average results is obtained by subtracting the monitoring office result from the original protein result (**see example**).
- (3) Range Difference. The difference between the original result and the monitoring result is determined for each individual sample monitored within a sample set. The range difference value that is plotted is the difference between the smallest and the largest difference observed in one set. If these two differences are of the opposite sign, add the magnitudes of the two numbers. The range difference is calculated and plotted on the range difference chart.
- (4) Plotted Results. The difference between the monitoring average result and the original result obtained from step 2 is plotted on the average difference chart. The positive differences (original results higher in protein than the monitoring results) are plotted above the center line. Negative differences (original results lower in protein than the monitoring results) are plotted below the center line. Results having no difference (original result is identical to monitoring result) are plotted on the center line.

Example of Plotting Control Chart				
Week Ending Date	Sample No.	Original Protein	ISE Protein	ORIG-ISE Difference
6/28/99	1	13.15	13.07	+0.08
6/28/99	2	10.18	10.54	-0.36
6/28/99	3	14.47	14.79	-0.32
6/28/99	4	12.56	12.23	+0.33
6/28/99	5	18.57	18.77	-0.20
Average		13.79	13.88	-0.09

In the example, the average difference between the service point and ISE is -0.09.

The range difference is calculated by determining the largest spread between the individual sample differences. In this example, the largest difference is +0.33 for sample No. 4 and -0.36 for sample No. 2. The spread or range between these two numbers is 0.69. Therefore, 0.69 is the range difference. Figures 1 and 2 illustrate average and range difference charts.

- d. Action Limits. Protein testing problems are indicated on the control charts either by a large difference between the average protein results or a consistent pattern of smaller differences on a series of average results. Three action limits are used for rapid identification of protein testing problems through interpretation of the control chart.

- (1) Absolute Limit (AL). This action limit is intended to identify excessive differences between results and indicates a potential protein testing problem. An absolute limit violation occurs if any plotted value is equal to or greater than the absolute limit lines.

Absolute limit lines are set at ± 0.20 percent protein for the average protein results. The range result absolute limit line is set at 0.60 percent protein for the protein range difference.

- (2) Tolerance Limit (TL). This action limit controls the number of consecutive data sets with a large difference between original and monitoring results but not so large as to exceed the absolute limit.

An average difference limit violation occurs if two consecutive data sets are either equal to or above the upper tolerance limit line or both equal to or below the lower tolerance limit line. The average difference tolerance limits are set at ± 0.15 percent protein.

A range difference tolerance limit violation occurs if two consecutive data sets are equal to or greater than the tolerance limit. The range difference tolerance limit is set at 0.40 percent protein.

- (3) Run Limit. This action limit controls the number of consecutive comparisons which are all above or all below the average difference chart center line (CL). A violation occurs if four out of five consecutive results are either all above or all below the CL, and the average difference from the center line for these four results exceed 0.10 percent protein. Run limits do not apply to the range difference chart.

5.7 COLLABORATIVE CHECK SAMPLES AND SPECIAL STUDIES

- a. Collaborative Check Samples. Collaborative check samples may be initiated by ISE for cross-checking other data. ISE will select and send enough of each sample selected so that specified service points can retain a portion for rechecking purposes. Upon receipt, participants must complete the analysis and report the data within 5 working days.

Use the forms provided with the samples to report analysis results. Retain a copy of the completed form and return the original immediately to ISE. Results from all locations included in the collaborative study will be compiled by ISE and reported to participating field offices and specified service points.

- b. Special Studies. ISE may initiate and conduct special studies. These studies are designed for a specific purpose (i.e., resolving differences either within or between markets, evaluating calibration performance, or updating the calibrations). When special studies are initiated by ISE, it is required that all participants (as designated by ISE) respond with utmost priority, as these are normally of an urgent nature and an expedient resolution of the problem is essential. Because ISE will not be routinely monitoring soybean or corn file samples, the field will be required to provide ISE with samples for monitoring and updating the calibration. Periodically throughout the year, ISE will contact selected field offices in soybean and corn markets and request that ten randomly selected soybean samples be provided to monitor calibration performance.

During harvest, ISE will request those field locations providing corn and soybean NIRT services to collect samples within specified ranges of protein, oil, and starch for use in updating the calibrations. Prior to harvest, ISE will notify the field of the types and numbers of samples needed and instructions for shipping them to ISE.

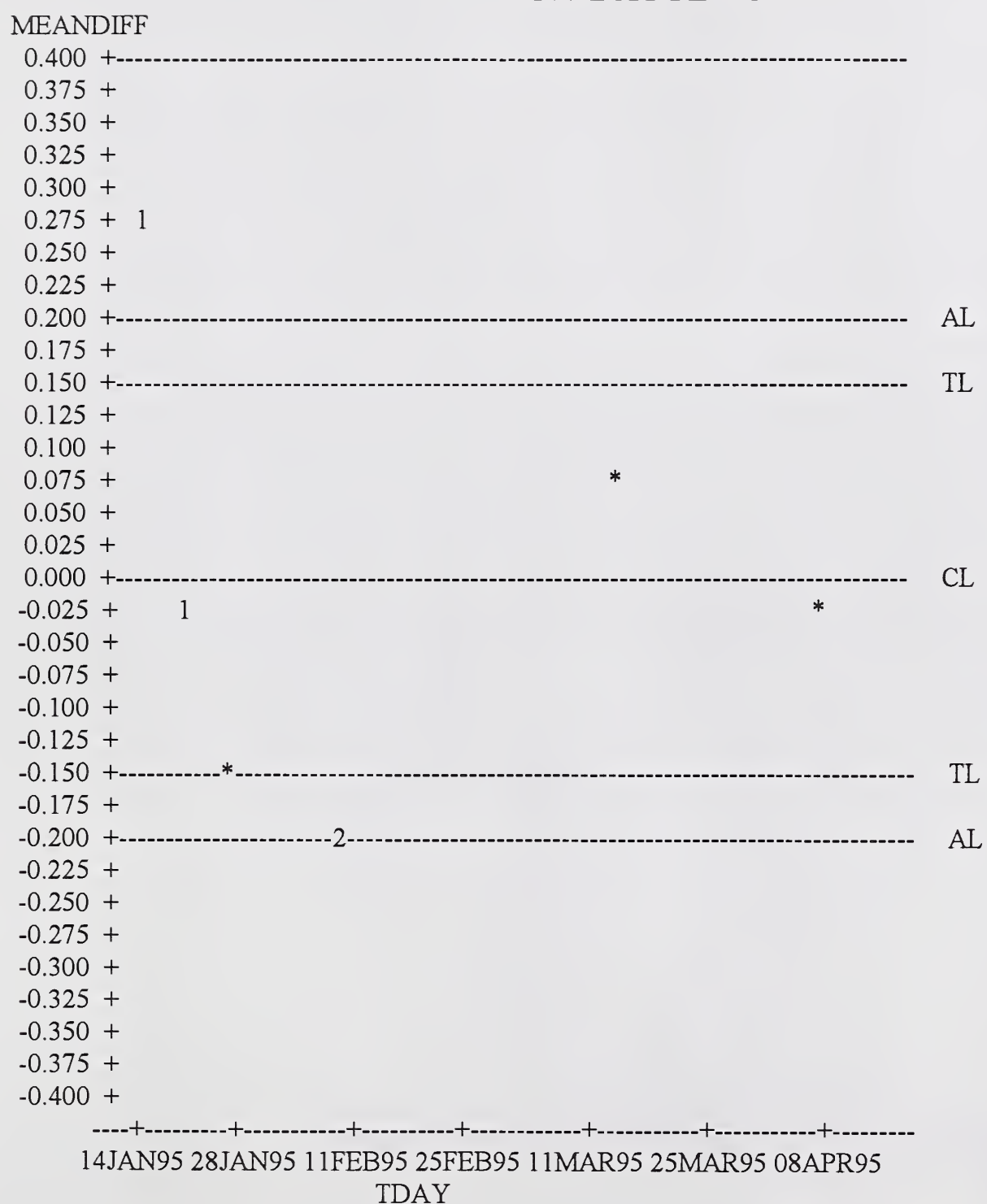
5.8 INTERMARKET SAMPLE EXCHANGE

An intermarket sample exchange helps isolate protein and/or oil differences between inspection points. Protein and oil testing laboratories will determine protein and oil results on separate portions obtained from the same sample. Protein and oil results are then compared to determine if any significant differences exist.

There are no restrictions as to which offices may exchange samples. Specified service points are encouraged to exchange samples with other specified service points and field offices for the purpose of resolving intermarket inspection differences. A copy of the results of the exchange must be provided to the field office and/or ISE for review if they were not participants in the exchange.

Figure 1

AVERAGE DIFFERENCE BETWEEN FIELD LOCATION AND ISE
180-DAY PERIOD



AL = Absolute Limit

TL = Tolerance Limit

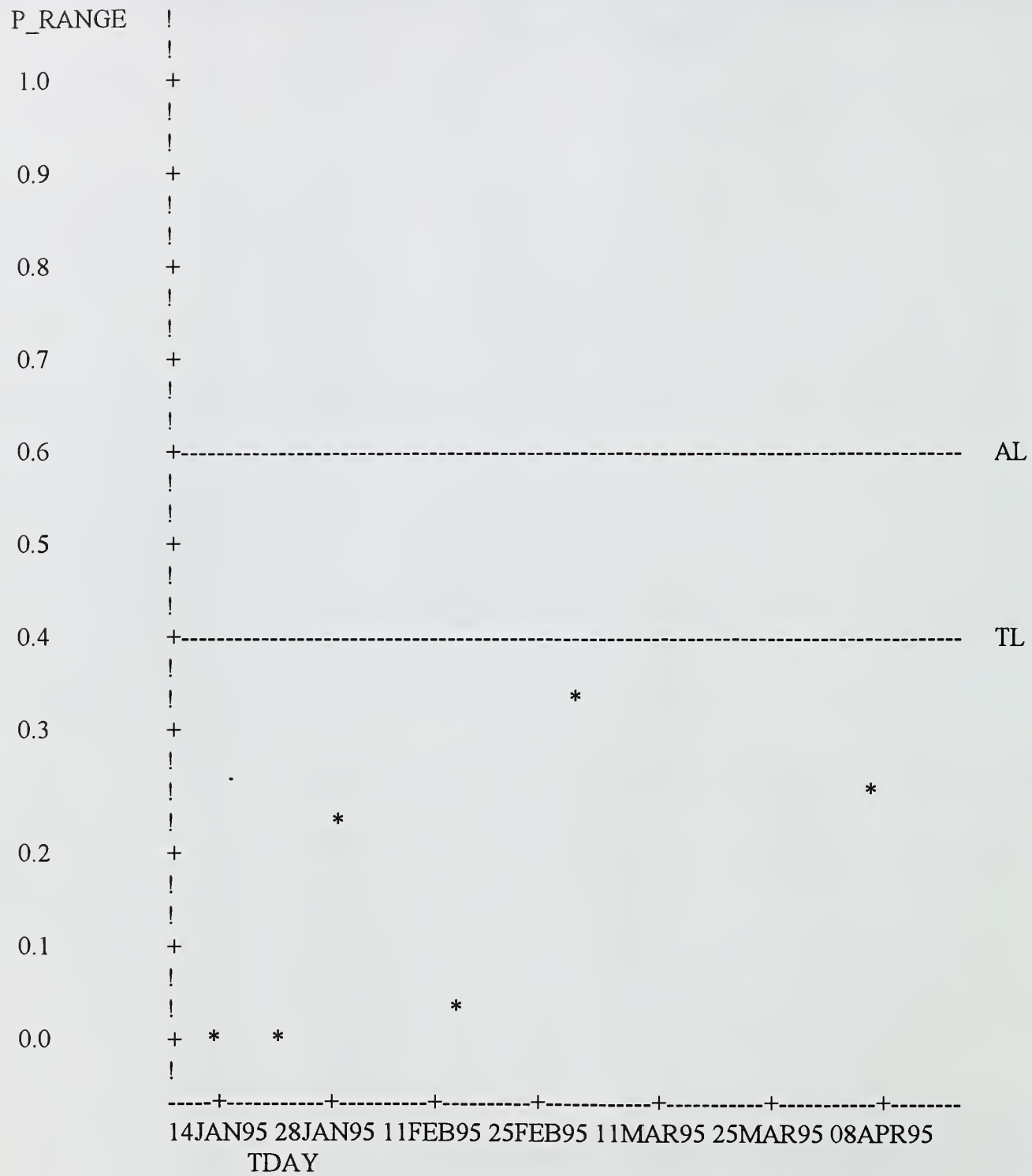
CL = Center Line

* = Average of 5 samples

1,2,3, or 4 = Number of
samples averaged

Figure 2

RANGE DIFFERENCE BETWEEN FIELD LOCATION AND ISE
180-DAY PERIOD



AL = Absolute Limit

TL = Tolerance Limit

